

STUDY OF DUCTILE COATINGS FOR THE OXIDATION  
PROTECTION OF COLUMBIUM & MOLYBDENUM ALLOYS

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A CORPORATE DIVISION OF TEXAS INSTRUMENTS INCORPORATED

### ABSTRACT

Air flow versus weight loss rate data on pure platinum has been extended to an air flow rate of 960 ipm at 2550 F. Increases in flow rate are seen to have a diminishing effect on weight loss rates at higher and higher flow rates.

Diffusion studies were performed on platinum-2.5% palladium alloy clad FS-85 and TZM at 2550 F. The diffusion appeared to be transgranular and very rapid for both the TZM and the FS-85 base samples. It is expected that these studies will be extended to include platinum and platinum-10% rhodium alloy coatings during the next report period.

Layers of gold, rhenium, aluminum, and boron nitride were deposited on refractory base metal for diffusion barrier studies. Evaluations of the samples containing aluminum and boron nitride have initially indicated that these materials will not be useful for diffusion barriers.

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## I. INTRODUCTION

The objective of this program is to explore the potential for development of coatings which will afford oxidation protection for 100 hours on columbium and molybdenum alloys at temperatures up to 3000 F. Also, these coatings should be ductile or, at least, flexible, and must not significantly degrade the mechanical properties of the alloys.

One molybdenum and one columbium alloy were selected for the investigations. They are, respectively, TZM (Mo-.5Ti-.08Zr-.02C) and FS-85 (Cb-28Ta-10W-1Zr). These alloys are considered to be two of the more promising refractory metal alloys.

Platinum and platinum-10% rhodium alloy were chosen as coating materials on the basis of oxidation resistance, ductility, and high melting point.

The efforts of this program in the first four bimonthly periods (Ref. 1) included:

1. A review of pertinent literature.
2. Selection and ordering of materials.
3. Setting up equipment for preparation and testing of samples.
4. Oxidation tests on coating materials.
5. Preparation and testing of coating-refractory base metal composites.
6. Preparation of samples containing diffusion barriers.

The work of the fifth bimonthly period has included:

1. A final oxidation test on coating metal.
2. Investigation of the coating-base metal interdiffusion process.
3. Preparation and evaluation of samples containing diffusion barriers.

A detailed discussion of this work is presented in the following paragraphs.

## II. OXIDATION TEST ON PLATINUM

Most of the potential areas of use for platinum coatings require high flow rates, e.g., turbine blades. Because of this, it was felt that the previously determined flow versus oxidation rate data should be extended to the limit of the present test setup - about 960 ipm.

Since most of the testing in the course of this program has been at 2550 F, that temperature was selected for the study.

Data for 24, 240, and 480 ipm had already been obtained in past report periods (Ref. 1).

A plot of all of the flow versus oxidation rates determined on platinum at 2550 F is presented in Fig. 1.

At higher and higher flow rates the effect of flow rate on oxidation rate is seen to diminish. This may be attributed to the rate of formation of oxide vapor playing a more important role at high flow rates than do the availability of oxygen for reaction and the removal of the vapor.

## III. COATING-BASE METAL DIFFUSION STUDIES

### A. Objective

The primary concern of this particular investigation was to obtain more information concerning the diffusion process associated with failure of coating-base metal composites in high temperature oxidation.

### B. Materials

It was planned that the diffusion studies would be made first with platinum coatings and then with platinum-10% rhodium alloy coatings. However, a new batch of platinum which had been obtained for these studies was found to contain about 2.5% palladium.

If this platinum-palladium alloy was to be considered unusable, a delay of at least several weeks would have been necessitated before pure platinum replacement material could be obtained. Normally such a wait would not be necessary; however, in the past few months platinum has become more and more difficult to acquire. Its scarcity is due primarily to Russia's removal of her platinum from the free market because of her present high rate of internal consumption for new chemical plants. As South African production is being increased and the Russian platinum is expected to return to the free market within a short period of time (Ref. 2) this situation is only temporary.

In view of the possible delay, the probability of obtaining useful results from the platinum-palladium alloy was given considerable attention.

First of all, palladium metal is reported to have a weight loss rate in air approximately equal to that of platinum at 2370 F, but with increasing temperature the rate increases well above that of both platinum and rhodium due to its high vapor pressure (Ref. 3). It is a very ductile metal but has been previously ignored in this program because of three of its properties:

1. Lowest melting point of the platinum metals - 2830 F.
2. High volatility - its weight losses in nitrogen at 2730 F are about 5 times as great as for platinum in air at that temperature (Ref. 4).
3. Permeability to oxygen - it has an oxygen solubility of 0.63% at 2200 F.

However, as it was believed that small percentages of palladium in platinum should have little effect on its melting point, volatility or oxygen permeability, the alloy was included in this diffusion study.

It should be noted that work using the platinum-palladium alloy has resulted in the development of improved test procedures.

It is expected that the results of tests with the platinum-palladium alloy will be compared with later tests with platinum and platinum-10% rhodium alloy to indicate whether or not palladium has any value as an alloying addition.

#### C. Preparation and Testing of Samples

The platinum-2.5% palladium alloy was roll bonded to TZM and FS-85, and samples 1-inch by 3/4-inch were cut from the bonded material. The edges of these samples were then undercut with acid and hot press bonded together, "sealing in" the base metal. The hot press bonding setup is shown in Fig. 2 and the finished sample in Fig. 3.

Three samples - (1) platinum-2.5% palladium alloy clad TZM, (2) platinum-2.5% palladium alloy clad FS-85, and (3) pure platinum - were tested simultaneously in each experiment, such that the effects attributable to the various variables could be more easily discerned from one another.

A single flow rate - 240 ipm, and test temperature - 2550 F, were used in each experiment. The tests were performed both in nitrogen and in air.



It was expected that a comparison of the samples tested in nitrogen with those tested in air would provide a better view of the effect of oxidation on outward diffusing refractory metal.

Photographs of each sample were taken at 1-hour intervals during testing in order to record the surface changes which occurred. Also, weight changes were determined after each of these 1-hour cycles.

Prior to testing, each sample was heated at 2550 F for about 3 minutes to remove surface contamination which might volatilize or burn off and lead to excessively high weight loss measurements in the initial test period.

For testing, the samples were positioned next to one another and about 1/2 inch apart in an alumina combustion boat, with the Pt-2.5% Pd/FS-85 sample furthest upstream and the Pt-2.5% Pd/TZM sample furthest downstream.

#### D. Results of Surface Examination

Figs. 4 through 21 show the surface conditions of the samples after 2, 6, and 12 hours of testing. As a result of thermal etching, the surface grain structure is clearly visible for each sample.

Some surface contamination is seen in the samples at lower magnification, and the TZM base sample heated in nitrogen (Figs. 9, 15, and 21) also shows a rolled-in particle of impurity at the higher magnifications. The general surface conditions obtained in testing these samples have been confirmed by repeat tests on additional samples.

The appearance of water droplet-like spots on the bonded samples heated in air led to additional observations on unbonded platinum and platinum-2.5% palladium alloy which had been rolled at the same time and on the same mill as the bonded samples. This material had been, consequently, subjected to conditions which would cause similar surface pickup. Figs. 22 and 23 show the surfaces of those samples after 6 hours at 2550 F in a 240-ipm air flow. The surface imperfections termed "water droplets" are evident on both samples and therefore not related to base metal diffusion or to palladium content, but simply to contaminants rolled into the surfaces of the samples.

The cladding on the composite samples was about 4 to 5 mils thick, or about twice the thickness used on previously tested platinum and platinum-10% rhodium alloy clad samples.

The first indication that diffusion had reached the surface of the samples was evidenced in the formation of small particles

(probably crystals) on the surfaces of composites after the fourth hour of testing in nitrogen. By the sixth hour in nitrogen (Figs. 13 and 15) the particles had grown substantially and those on the TZM base sample were taking on a definite rod-like crystalline shape. The continued growth of these particles is seen in Figs. 19 and 21, after 12 hours of testing. The uniform distribution of particles, both by size and position, indicates that the diffusion was transgranular rather than intergranular.

In contrast to the samples tested in nitrogen, no particles developed on the composites tested in air. The FS-85 base sample had a surface appearance that paralleled that of platinum except for the few oxide-containing blisters that formed and began to rupture after about 7 hours of testing.

The surface grains in the cladding on the TZM base sample were seen to become gradually more and more irregular until after 12 hours they looked as though they had been "shot peened." Those convexities with the greater heights show up as black spots in the pictures due to focusing effects. A surprisingly large percentage of these occur in the grain boundaries. The cause of these surface irregularities is not yet known.

The only notable surface changes to occur on the pure platinum samples, during testing in nitrogen and air, were small grain boundary shifts. Atmosphere differences appeared to have very little effect.

#### E. Weight Changes During Testing

Figs. 24 and 25 show the weight losses for the three samples tested in air and nitrogen, respectively. The weight loss rates on the clad samples are obviously much higher than for pure platinum. The relatively high weight losses on the clad samples tested in nitrogen give some idea of the volatility of the palladium in the alloy.

For comparison with the clad material, two pieces of unclad platinum-2.5% palladium alloy were tested in a 240-ipm air flow at 2550 F and the weight loss results are presented in Fig. 26. The surface condition of one of these, Sample 1, was shown in Fig. 22 after 6 hours of testing. The weight losses for the samples are higher than obtained for bonded material; however, it should be remembered that, prior to testing, the bonded samples had been heated to red heat while sealing their edges; and, also, they had received a few minutes of clean-up heating at 2550 F. The unclad material received neither of these treatments and would, therefore, contain more palladium.

Initial metallographic examination of the coating-base metal interface in these samples has evidenced diffusion layers somewhat

similar to those obtained in past report periods (Ref. 1) with platinum and platinum-10% rhodium alloy coatings. A more detailed examination of these samples is considered desirable and will be conducted in the next report period.

#### 17. DIFFUSION BARRIER STUDIES

Four barrier materials - gold, rhenium, aluminum, and boron nitride, were included in the investigations of the fifth period. Each is discussed separately below.

##### A. Gold

In his work on platinum protected molybdenum, Rhys (Ref. 5) showed gold to be effective in retarding interdiffusion between the platinum and molybdenum at 2200 F. Although it has not as yet been verified, it is believed that the gold itself does not act to retard diffusion, but that it forms a compound with the molybdenum or with the molybdenum and platinum which acts as the barrier. Because of Rhys' findings, gold was selected as a material for further investigation as a diffusion barrier in this study.

Techniques had been previously developed for roll bonding gold to molybdenum; however, application of both gold and platinum to a refractory metal is a more difficult problem. Microscopically, small pockets of air are entrapped between platinum and gold during roll bonding and, as there is no means of escape, heating will cause the air to expand into numerous small blisters. Normally, the surfaces of the materials to be roll bonded are roughened prior to bonding. For bonding platinum and gold, however, it was found that blistering could be minimized by roll bonding smooth surfaces which had been cleaned by heating to about 1200 F in air.

Initially, FS-85 alloy has been used in the preparation of samples for tests with gold diffusion barriers. It was selected over TZM because its greater ductility makes it easier to work.

The platinum/gold/FS-85 samples that have been prepared will be sintered at approximately 1800 F for a hour prior to testing at 2550 F. The purpose of this sintering will be to form the expected gold intermetallic compound (auride), which is to act as a barrier to diffusion.

Platinum/gold/TZM samples will be prepared and tested with the FS-85 base samples during the next bimonthly period.

##### B. Rhenium

In the work of Passmore, et al (Ref. 6), rhenium was seen to be the most promising metallic diffusion barrier for use on refractory metals. Also, Deak (Ref. 7) found it to be a useful diffusion barrier between molybdenum and platinum for grid wire in electron

tubes where the temperature may be as high as 2200 F.

Samples of FS-85, coated with an estimated 0.05 to 0.1 mils of rhenium, were obtained from Summit Finishing Company of Thomaston, Connecticut. The coatings were applied electrolytically from aqueous solution.

Observed with the unaided eye, these coatings were seen to have a dark gray dull finish - possibly due to surface oxidation. Metallographic examination of the coating-base metal interface has proved difficult. The most important problem has been protection of the edges of the samples during polishing, such that the very thin rhenium coating could be observed with good resolution. Attempts to electroplate nickel and/or copper onto the coatings, to protect the outer edge of the rhenium during polishing, have not been successful. The electroplates had poor adherence and pulled away from the rhenium during mounting. Further work is planned during which the material will be given a hydrogen anneal to clean up any surface oxidation prior to additional metallographic work or preparation of test samples.

#### C. Aluminum

Aluminum, as such, would not be expected to act as a diffusion barrier for the given coating-base metal combinations and test conditions. However, the aluminides it forms with the two base metal alloys and the oxidation products of these intermetallics are high melting, appreciably stable compounds and were, therefore, deemed worthy of consideration.

Three experiments were conducted using aluminum:

1. Sintering aluminum clad FS-85 in air to produce an oxide barrier coating.
2. Sintering aluminum clad FS-85 in argon and then in air to produce an oxide barrier coating.
3. Sintering platinum/aluminum/FS-85 composites to produce an aluminide barrier layer between the platinum coating and FS-85 alloy base metal.

In the first experiment, sintering at 1200 F for 10 minutes, followed by sintering for 10 minutes at 1300 F produced a number of base metal oxide "break throughs" in the cladding. The initial aluminum cladding thickness for this sample was about 3 mils.

For the second experiment a 3-mil thickness of cladding was again used. The first sintering was done at 1900 F in argon for 1/2 hour to form the aluminide before any oxidation. The surface of the sample given this treatment appeared relatively smooth and well reacted. However, on reheating at the same temperature in air, several failures occurred in the coating and the base metal was subjected to oxidation and embrittlement.

The Pt/Al/FS-85 composites for the third experiment were prepared by roll bonding and sintering at 1200 F for 1/2 hour in argon. The "as bonded" thicknesses were (1) platinum coating-2.3 mils, (2) aluminum-0.8 mils, (3) FS-85 base metal-14 mils. The inter-metallic produced by sintering was so brittle that the coating broke away from the base metal during handling.

In the studies described above aluminum showed little promise as a diffusion barrier material. It may be, though, that other preparation techniques will lead to more successful samples.

#### D. Boron Nitride

This material was selected for evaluation as a potential diffusion barrier on the basis of past experience with its chemical inertness (Refs. 8 and 9) and a previously developed "in-house" capability to apply it as a thin flexible coating on refractory metals.

Boron nitride coatings were applied to material for test samples through the decomposition of trichloroborazole on the surface of heated TZM and FS-85 alloy strip. The thickness of the boron nitride layer was estimated to be 1.5 microns.

Initially, sputtering appeared to be the most convenient method for application of platinum onto the boron nitride coated samples. Through sputtering the platinum could be deposited on freshly laid down boron nitride with a minimum exposure to atmospheric contaminants or handling that might produce surface damage. Using this process, however, it was found necessary to heat the samples to the melting point of platinum in order to make the platinum layers adherent.

Only a low deposition rate was attainable through the sputtering process and it was found necessary to add additional platinum through hot press bonding.

As reported in the last bimonthly report (Ref. 1), a sample was prepared with a 2-mil thickness of platinum (2 mils of platinum hot press bonded to 1 micron of sputtered platinum), a 1.5 micron thickness of boron nitride, and a 15-mil thickness of FS-85 alloy.

In flowing air at 2550 F this sample failed in just a few hours. This failure occurred through the formation and rupture of blisters at the edges of the sample and it was felt that this was an "edge protection" problem. However, metallographic examination has indicated the complete lack of any barrier to diffusion in the tested sample (as evidenced by the occurrence of diffusion layers), and a lack of edge protection apparently had little to do with the failure.

Metallographic examination of a platinum/boron nitride/FS-85 sample, which had not been tested, failed to reveal any boron nitride layer. It is important to note, however, that by virtue

of its thinness and softness, the boron nitride layer may be difficult to expose for metallographic examination.

On the basis of this preliminary investigation, then the Pt/BN/FS-85 samples do not look good. The ineffectiveness of the boron nitride in stopping diffusion may be attributed to one of two possible causes:


1. Degradation of the boron nitride layer due to incompatibility with the platinum and/or FS-85.
2. Loss of the layer through some process associated with the deposition of the sputtered platinum coating.

A number of samples have been prepared on which there is a clearly visible layer of boron nitride. Tests on these should substantiate whether or not boron nitride may be used as a barrier to coating-base metal diffusion.

A different type of sample configuration will be used for additional tests on samples containing boron nitride barrier layers. As shown in Fig. 27, it has an additional thickness of platinum at the edges to prevent any "edge protection" problem from arising. The components of the sample will be bonded together through the application of heat and pressure. A graphite clamp, Fig. 28, has been prepared to hold the composite, and expansion differences between this clamp and the sample should produce a pressure on the sample that increases with increasing temperature and causes bonding.


#### V. WORK PLANNED FOR THE NEXT PERIOD

1. Coating-base metal diffusion studies - to include electron microprobe analyses, microhardness traverses, and possibly X-ray diffraction studies of the sample surfaces.
2. Evaluation of diffusion barriers.

  
E. H. Girard, Project Engineer

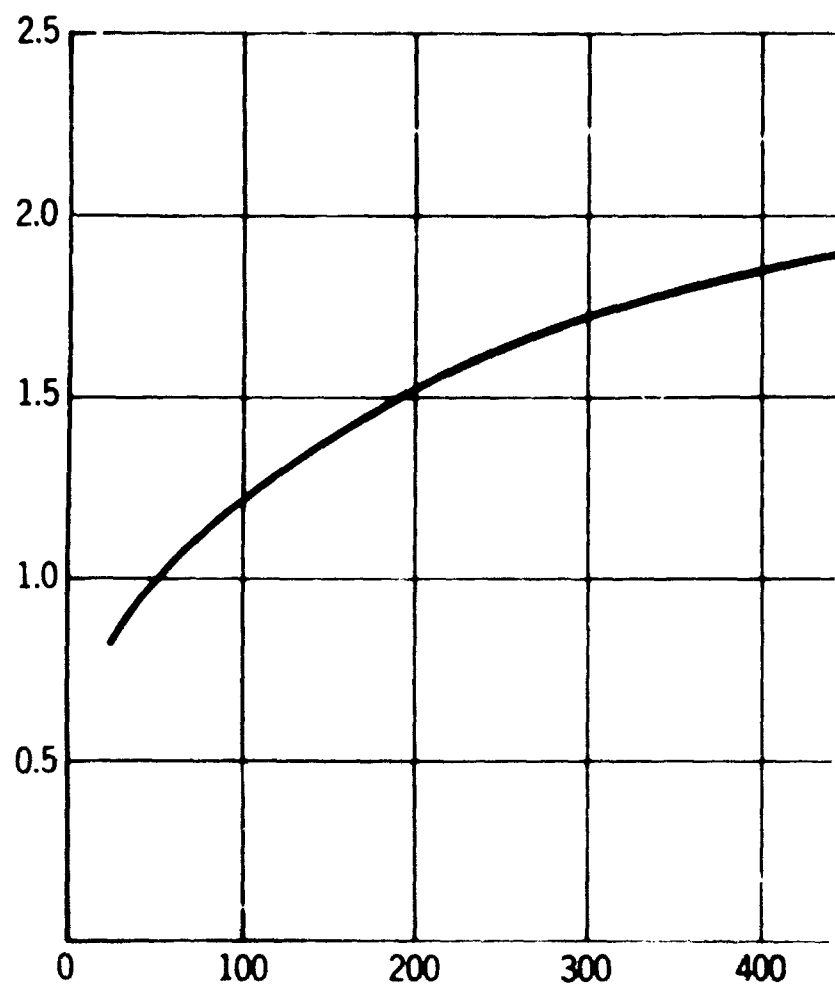
  
J. B. Buchinski, Program Manager

Approved:

  
Dr. T. S. Burkhalter  
Director, Research and Development  
Materials and Controls Division

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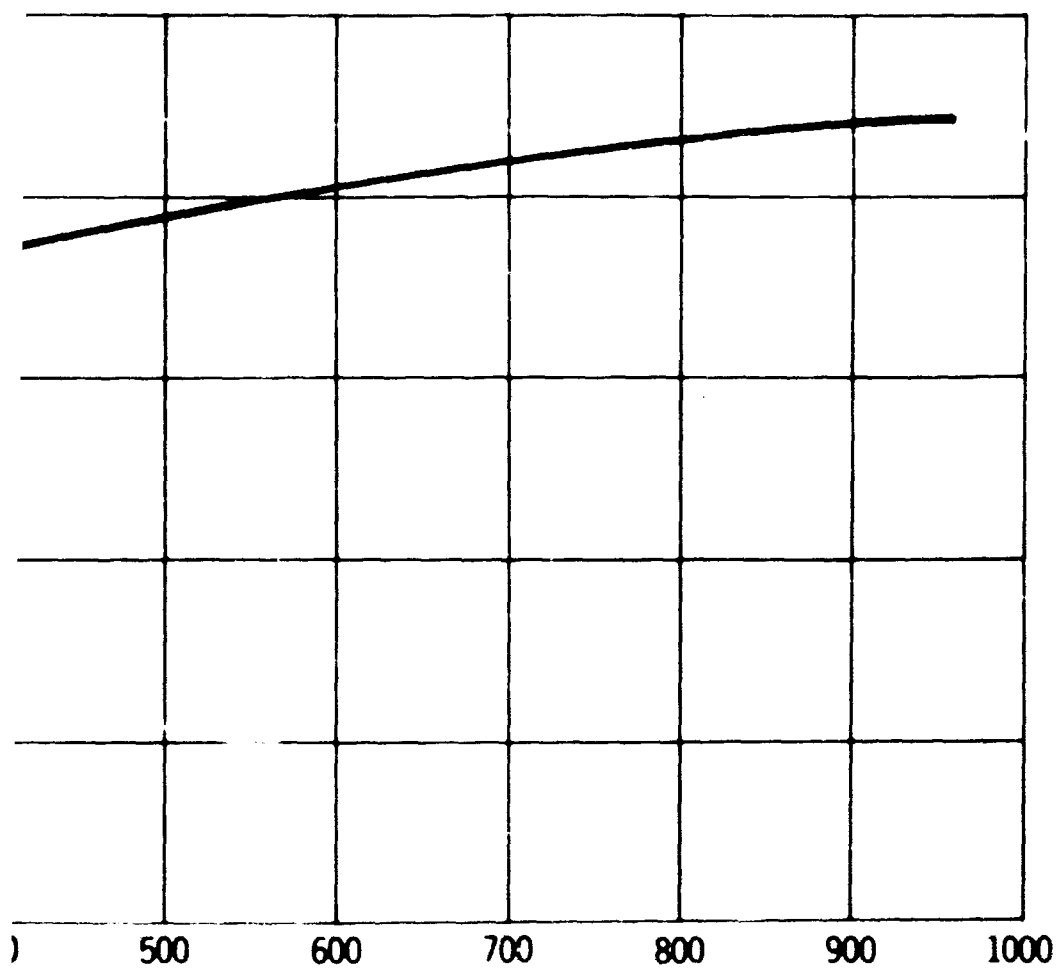
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OXIDATION RATE, MILS PER HOUR  $\times 10^3$ 

AIR I

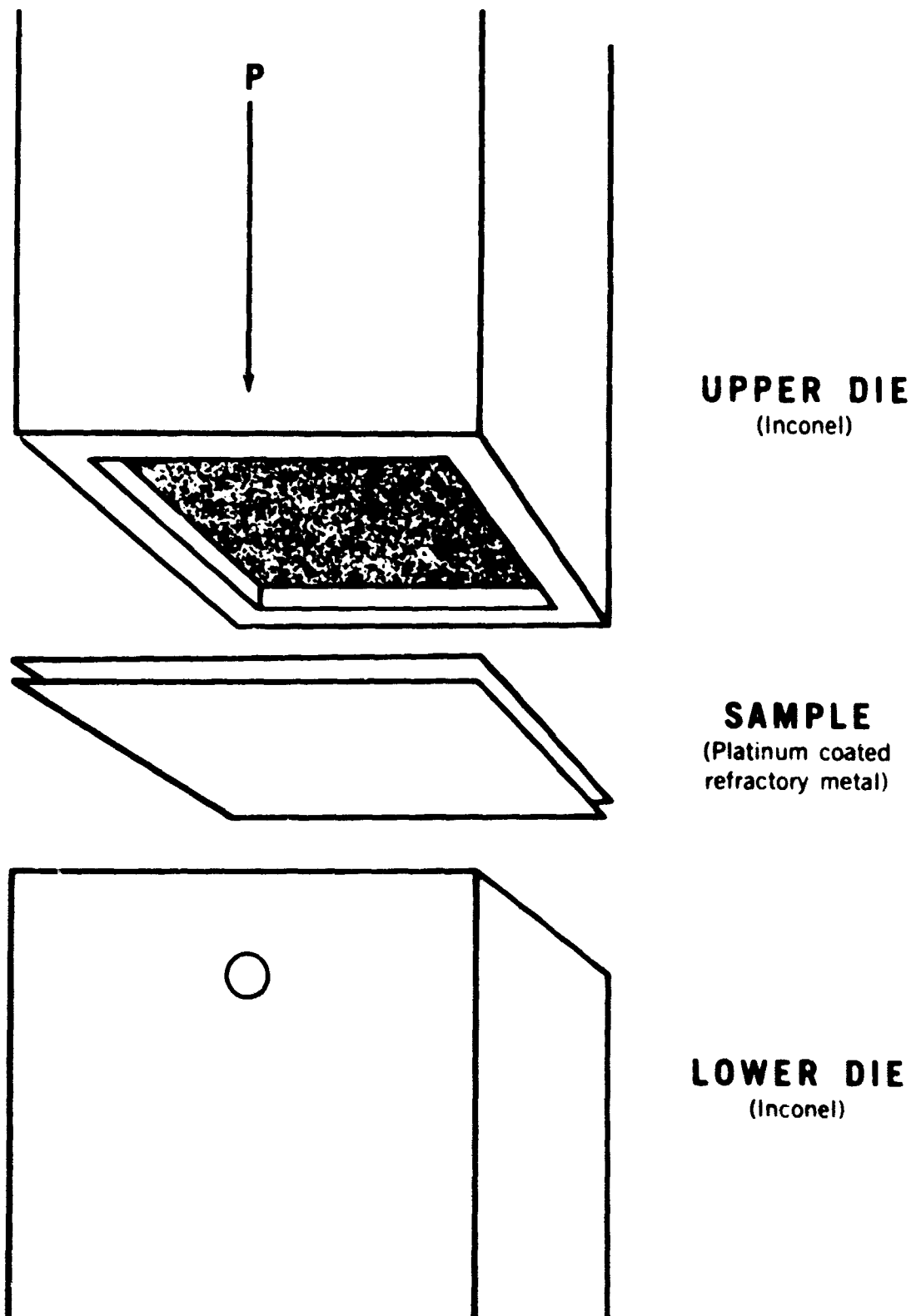
F  
Thickness loss rate for platinum



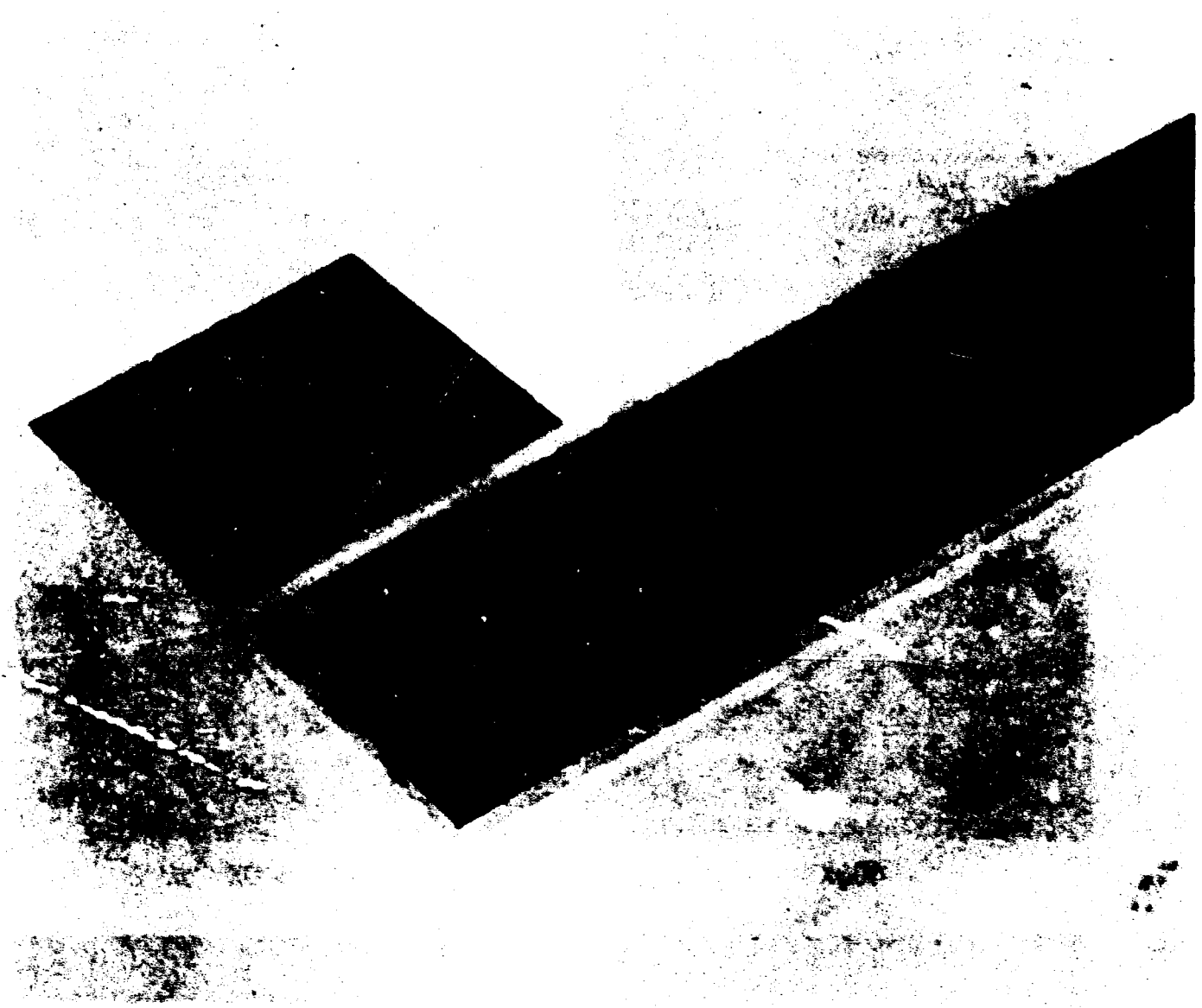


AIR FLOW RATE, IPM

**FIGURE 1**  
atinum versus air flow rate at 2550 F.



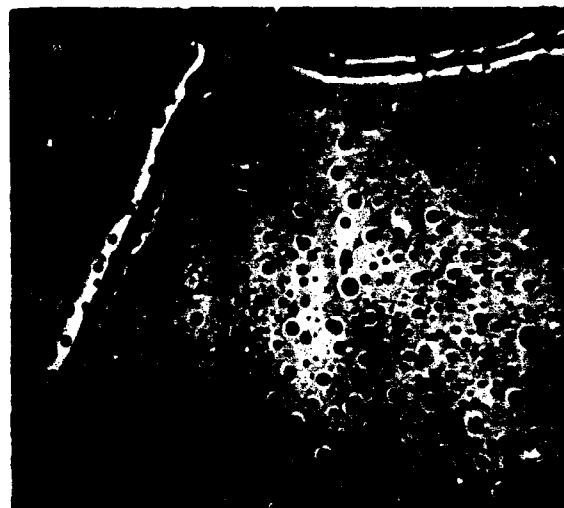
**FIGURE 2**  
Setup for sealing sample edges by Hot Press Bonding  
(Induction Heating Coil is not shown).



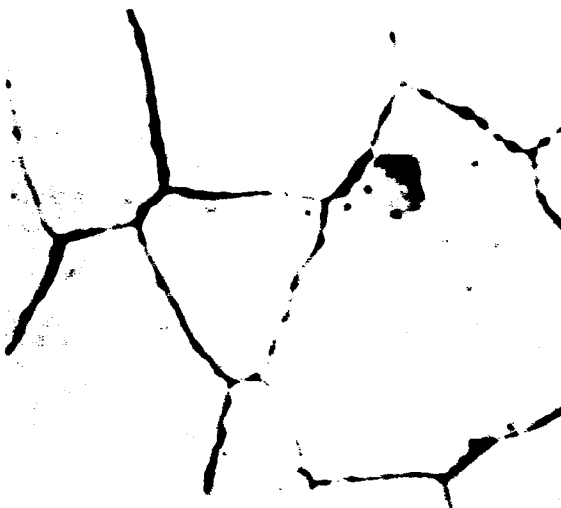
**FIGURE 3**  
Platinum coated refractory metal alloy test sample



1000X



1000X



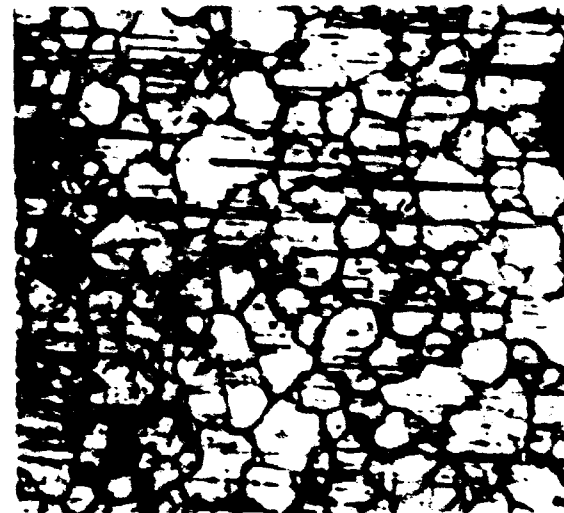
400X



400X



60X



60X

**FIGURE 6**

Surface condition of Pt-2.5% Pd alloy clad FS-85 after 2 hours at 2550 F in a 240 ipm air flow.

**FIGURE 7**

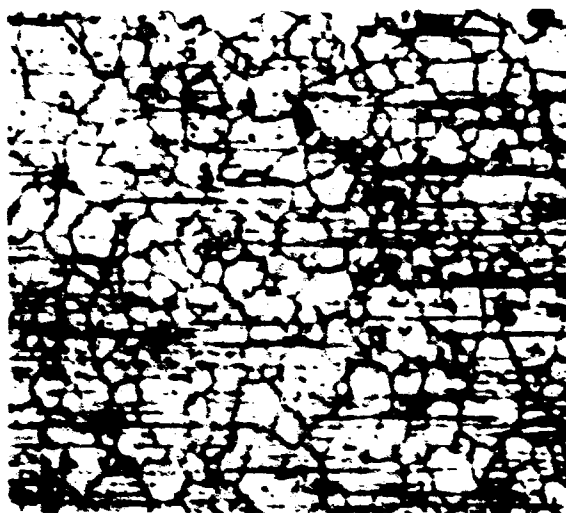
Surface condition of Pt-2.5% Pd alloy clad FS-85 after 2 hours at 2550 F in a 240 ipm nitrogen flow.



1000X



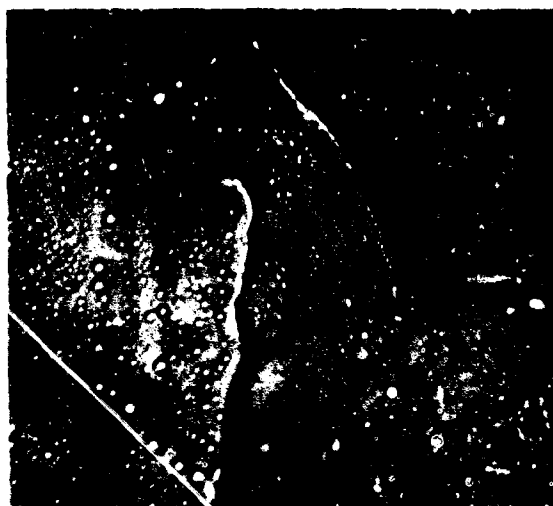
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60X

**FIGURE 8**

Surface condition of Pt-2.5% Pd alloy clad TZM after 2 hours at 2550 F in a 240 ipm air flow



1000X



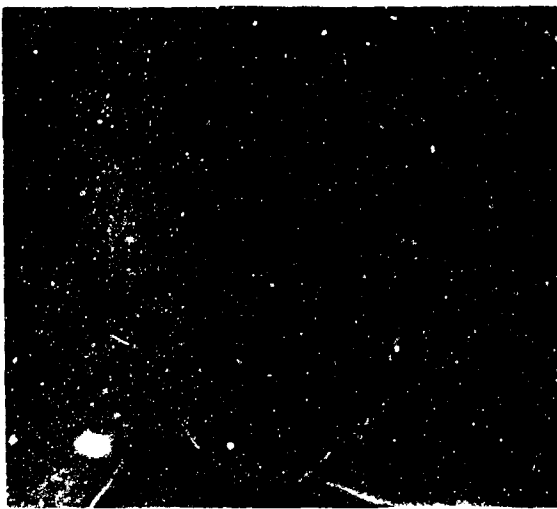
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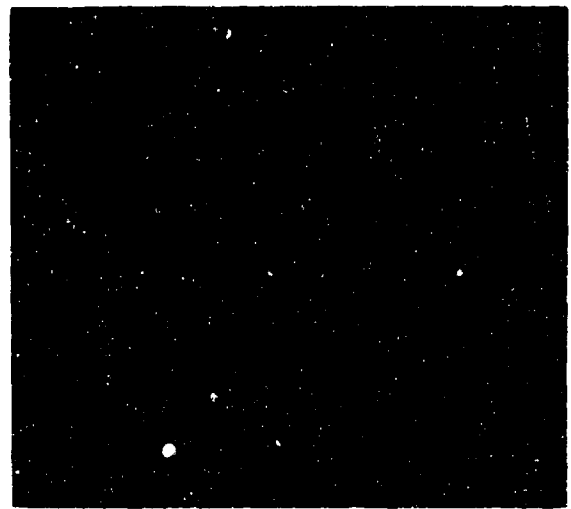
60X

**FIGURE 9**

Surface condition of Pt-2.5% Pd alloy clad TZM after 2 hours at 2550 F in a 240 ipm nitrogen flow



1000X



1000X



400X



400X



60X



60X

**FIGURE 10**

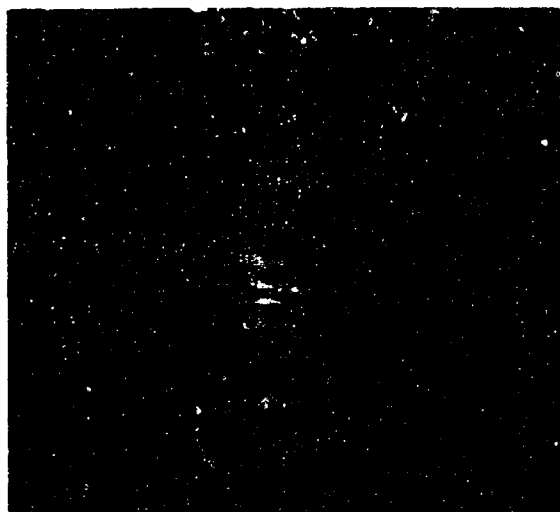
Surface condition of pure platinum after 6 hours at 2550° in a 240 ipm air flow.

**FIGURE 11**

Surface condition of pure platinum after 6 hours at 2550°F in a 240 ipm nitrogen flow.



1000X



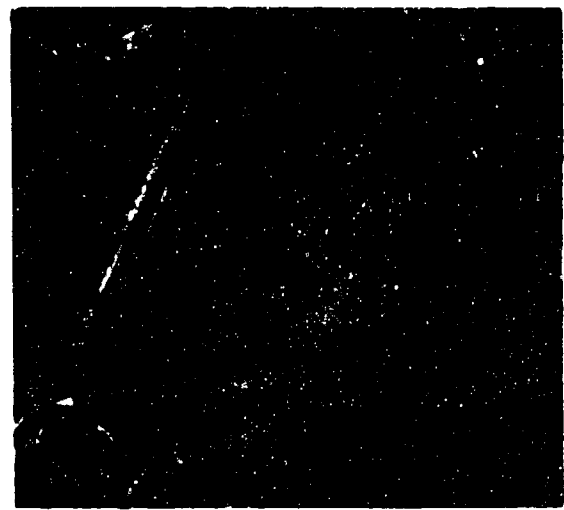
400X



60X

**FIGURE 12**

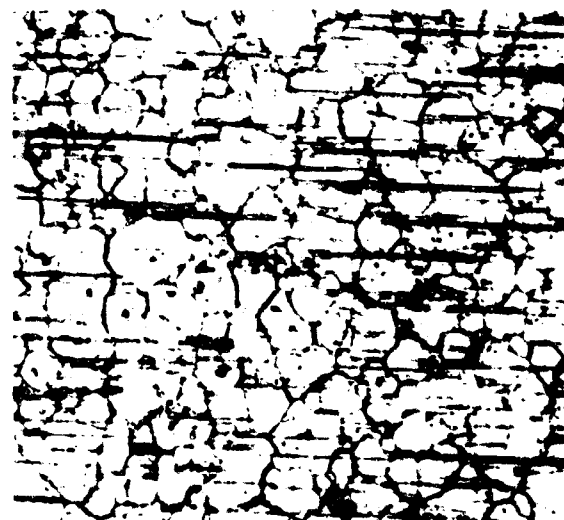
Surface condition of Pt-2.5% Pd alloy clad FS-85 after 6 hours at 2550°F in a 240 ipm air flow.



1000X



400X



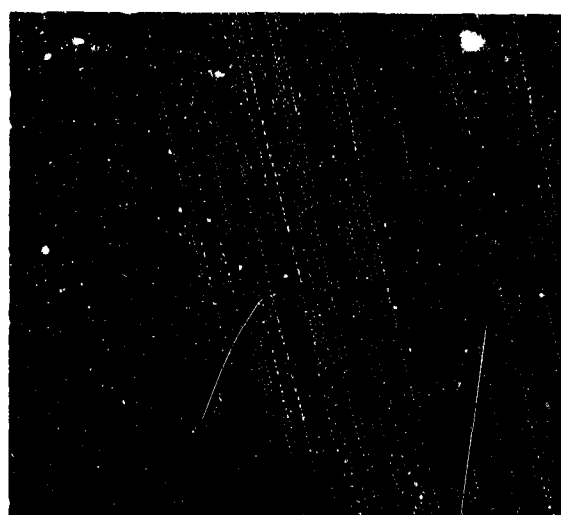
60X

**FIGURE 13**

Surface condition of Pt-2.5% Pd alloy clad FS-85 after 6 hours at 2550°F in a 240 ipm nitrogen flow.



1000X



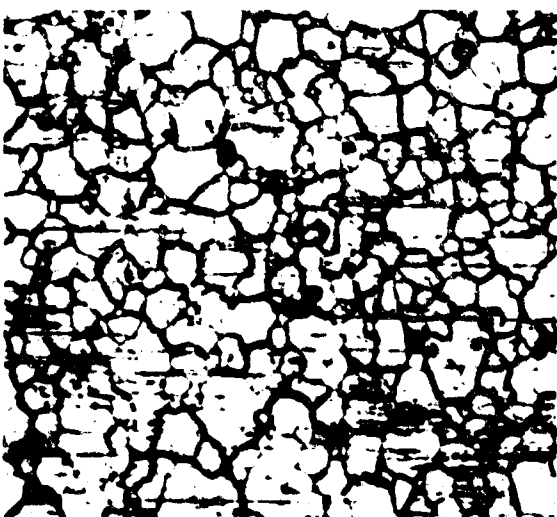
1000X



400X



400X



60X



60X

**FIGURE 14**

Surface condition of Pt-2.5% Pd alloy clad TZM after 6 hours at 2550°F in a 240 ipm air flow.

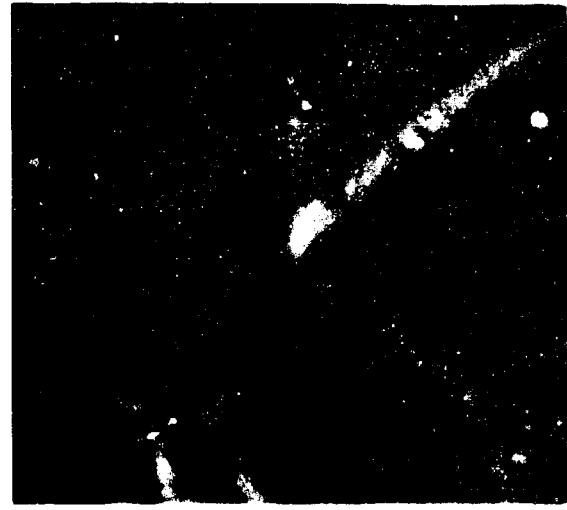
**FIGURE 15**

Surface condition of Pt-2.5% Pd alloy clad TZM after 6 hours at 2550°F in a 240 ipm nitrogen flow.





1000X



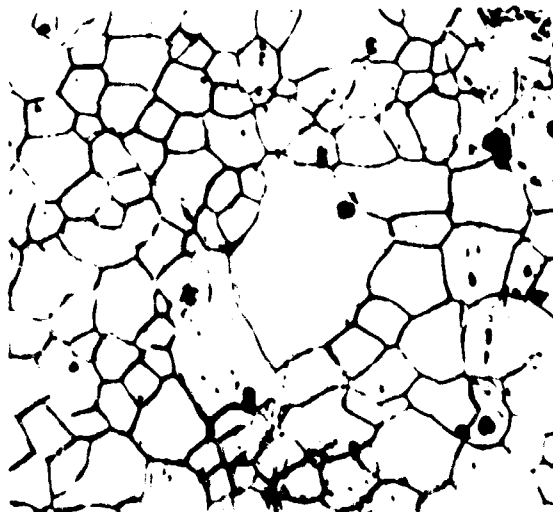
1000X



400X



400X



60X



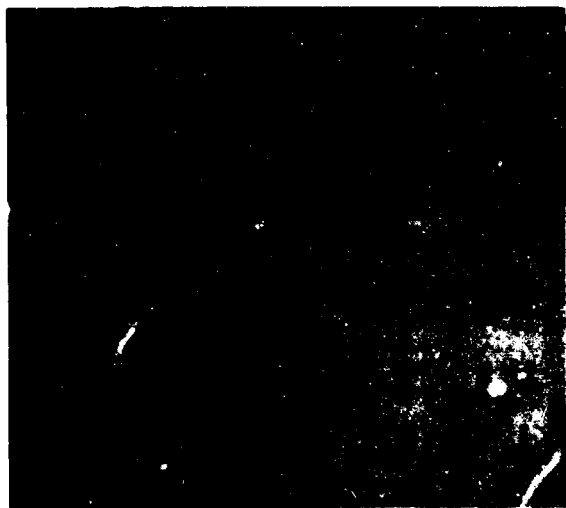
60X

**FIGURE 16**

Surface condition of pure platinum after 12 hours at 2550°F in a 240 ipm air flow.

**FIGURE 17**

Surface condition of pure platinum after 12 hours at 2550°F in a 240 ipm nitrogen flow.



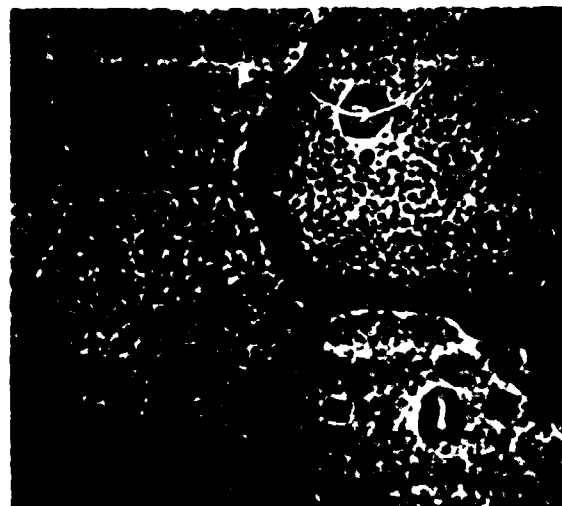
1000X



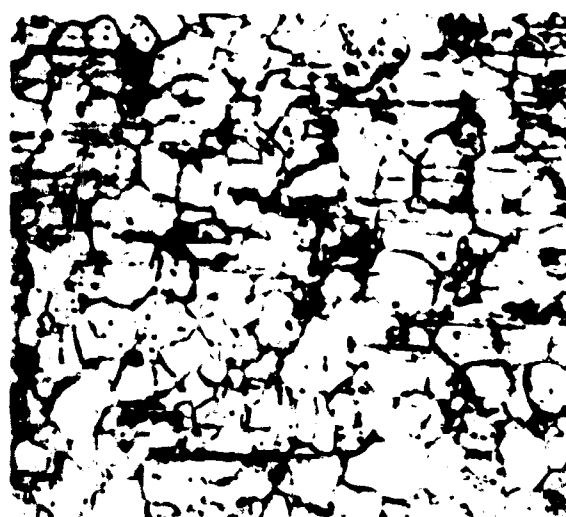
1000X



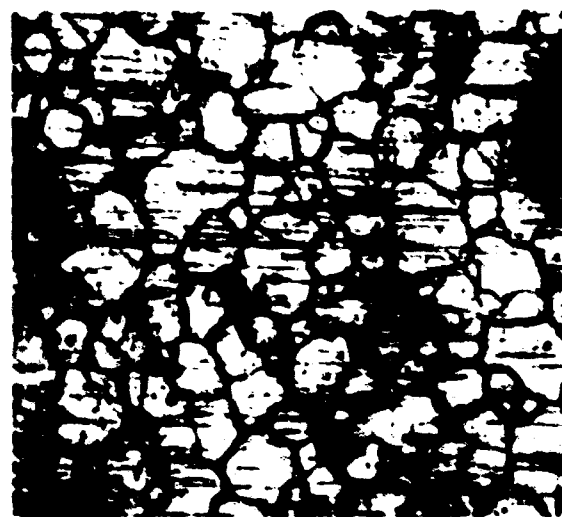
400X



400X



60X



60X

**FIGURE 18**

Surface condition of Pt-2.5% Pd alloy clad FS-85 after 12 hours at 2550 F in a 240 ipm air flow.

**FIGURE 19**

Surface condition of Pt-2.5% Pd alloy clad FS-85 after 12 hours at 2550 F in a 240 ipm nitrogen flow.



1000X



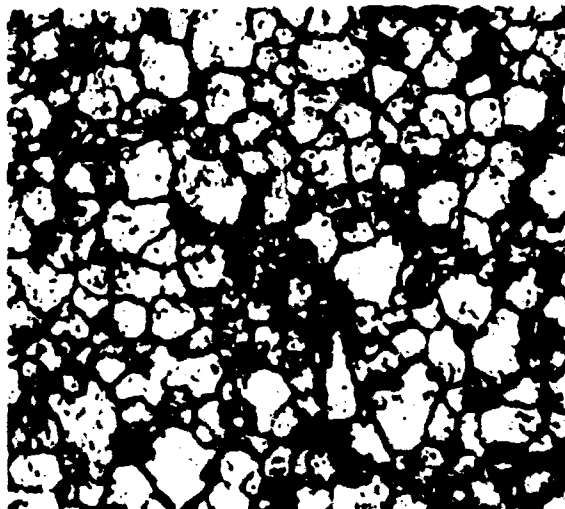
1000X



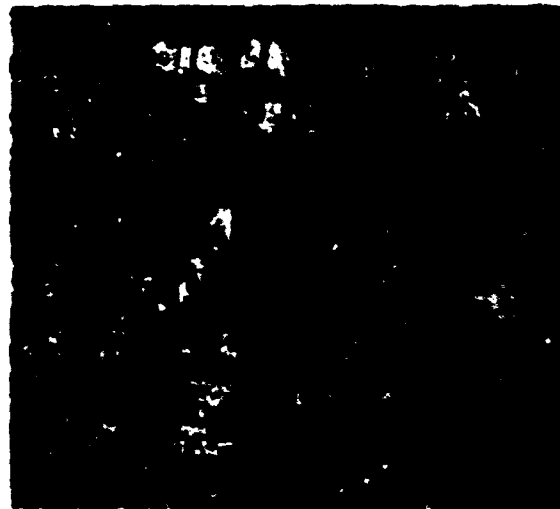
400X



400X



60X



60X

**FIGURE 20**

Surface condition of Pt-2.5% Pd alloy clad TSM after 12 hours at 2550 F in a 240 ipm air flow.

**FIGURE 21**

Surface condition of Pt-2.5% Pd alloy clad TSM after 12 hours at 2550 F in a 240 ipm nitrogen flow.



1000X



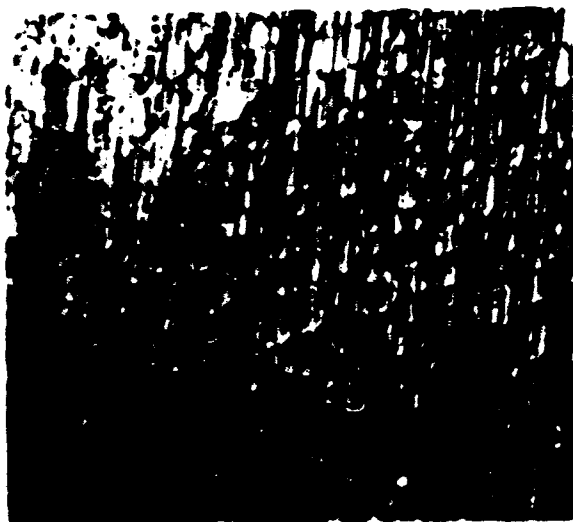
1000X



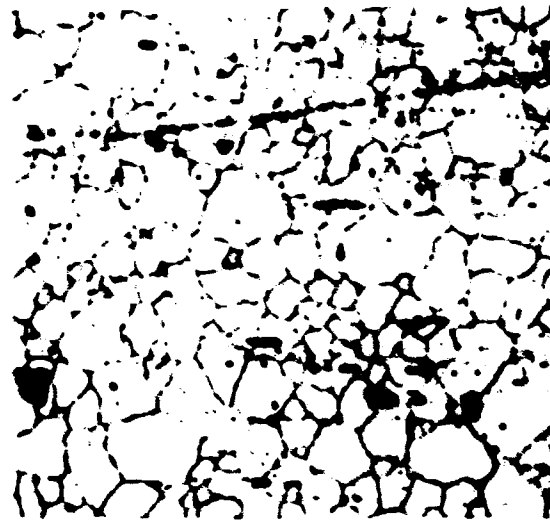
400X



400X



60X



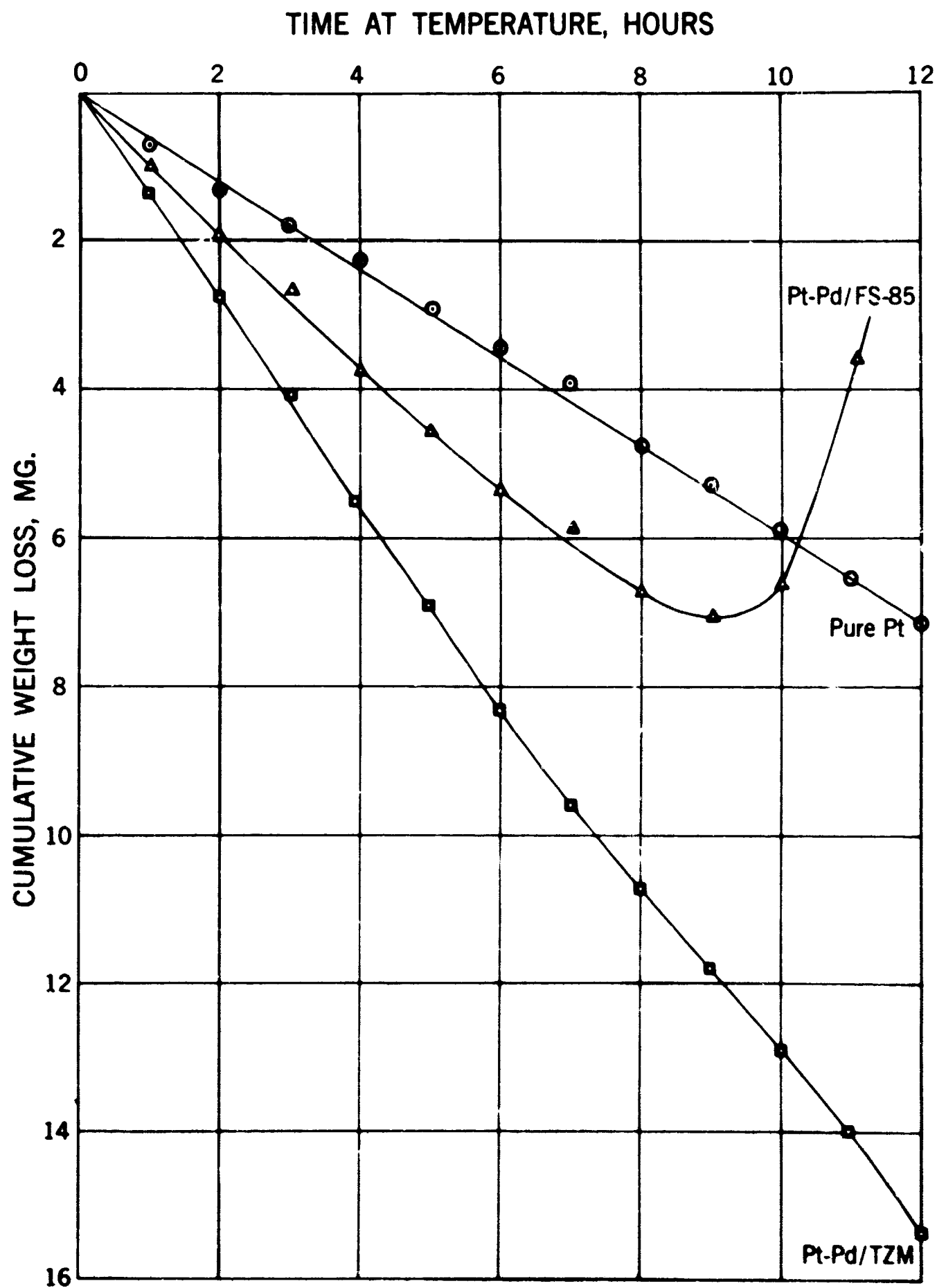
60X

**FIGURE 22**

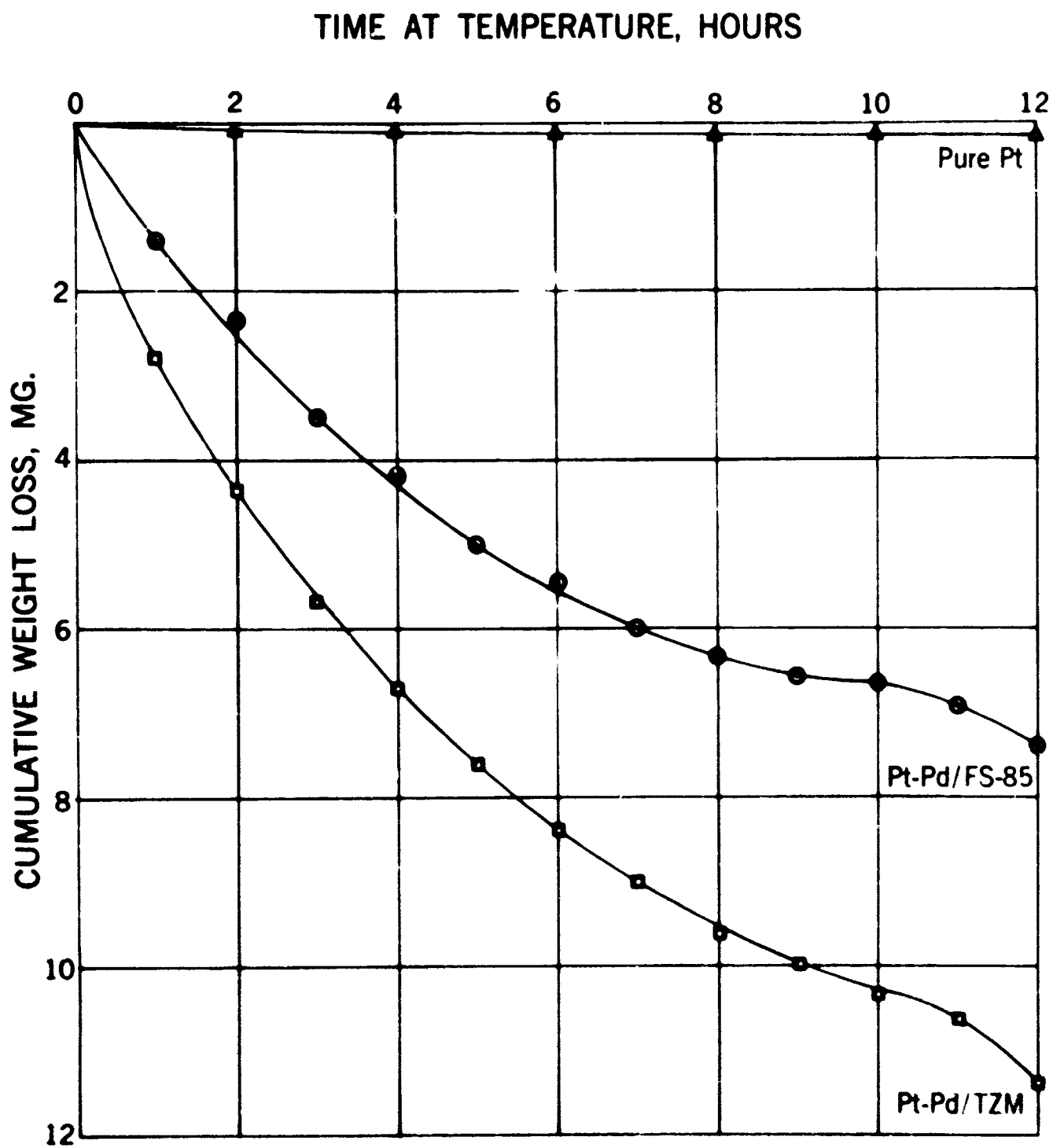
Surface condition of a piece of platinum-25% palladium alloy after 6 hours at 2550 F in a 240 ipm air flow.

**FIGURE 23**

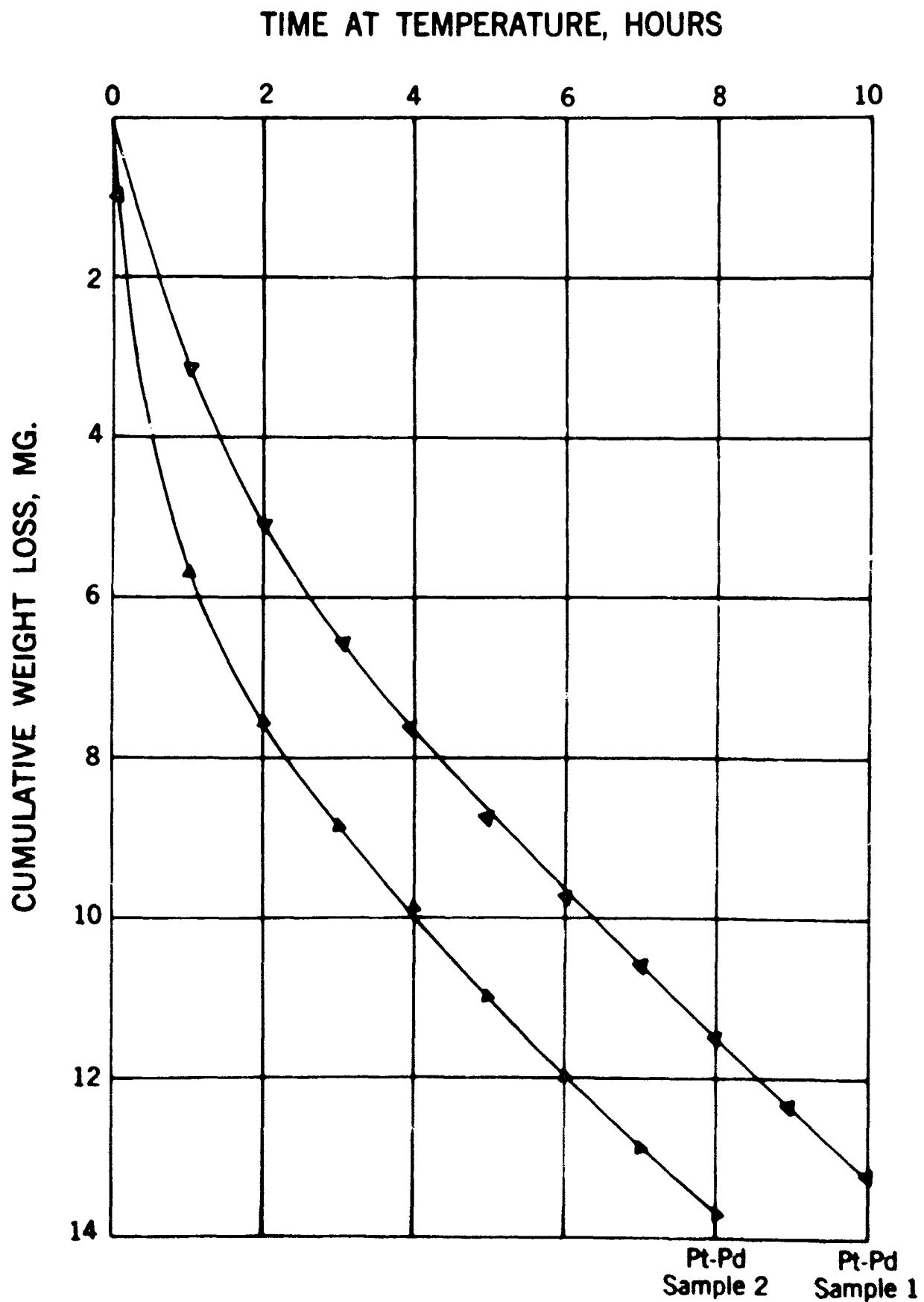
Surface condition of a piece of pure platinum after 6 hours at 2550 F in a 240 ipm air flow.



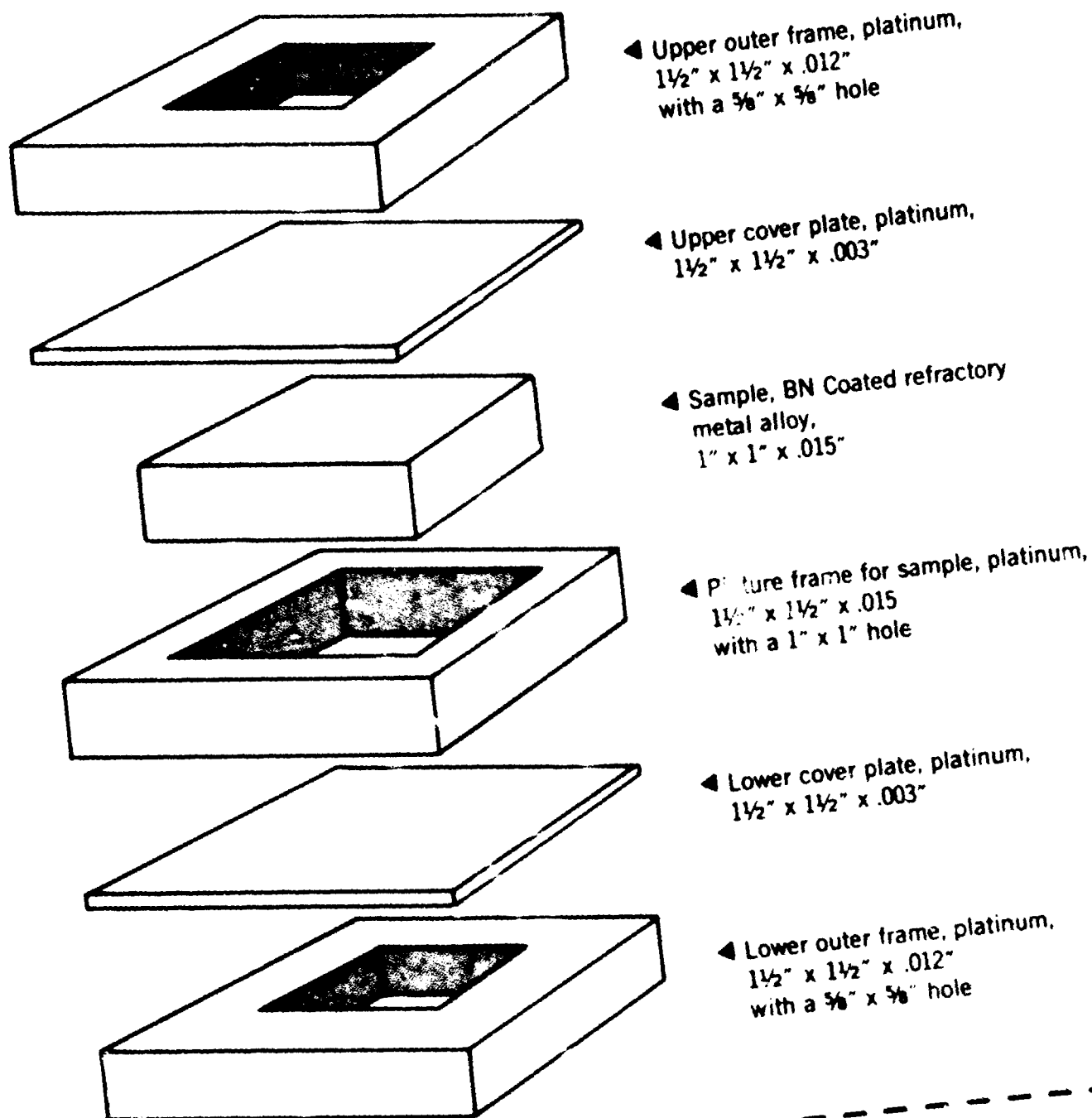
**FIGURE 24**  
 Weight losses for platinum-2.5% palladium alloy clad FS-85 and TZM,  
 and for pure platinum in a 240 IPM air flow at 2550°F.



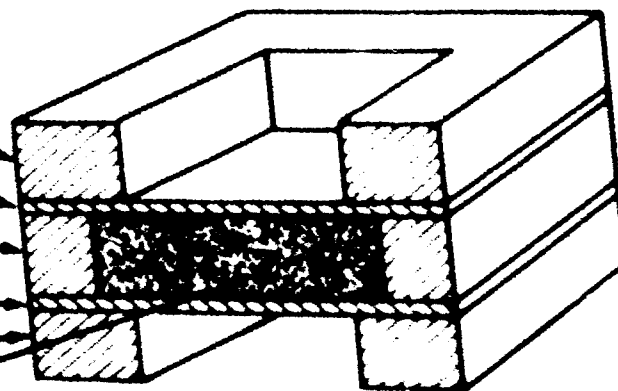
**FIGURE 25**  
Weight losses for platinum-2.5% palladium alloy clad FS-85 and TZM,  
and for pure platinum in a 240 IPM nitrogen flow at 2550°F.



**FIGURE 26**  
Weight losses for two platinum-2.5% palladium alloy samples  
in a 240 IPM air flow at 2550°F.

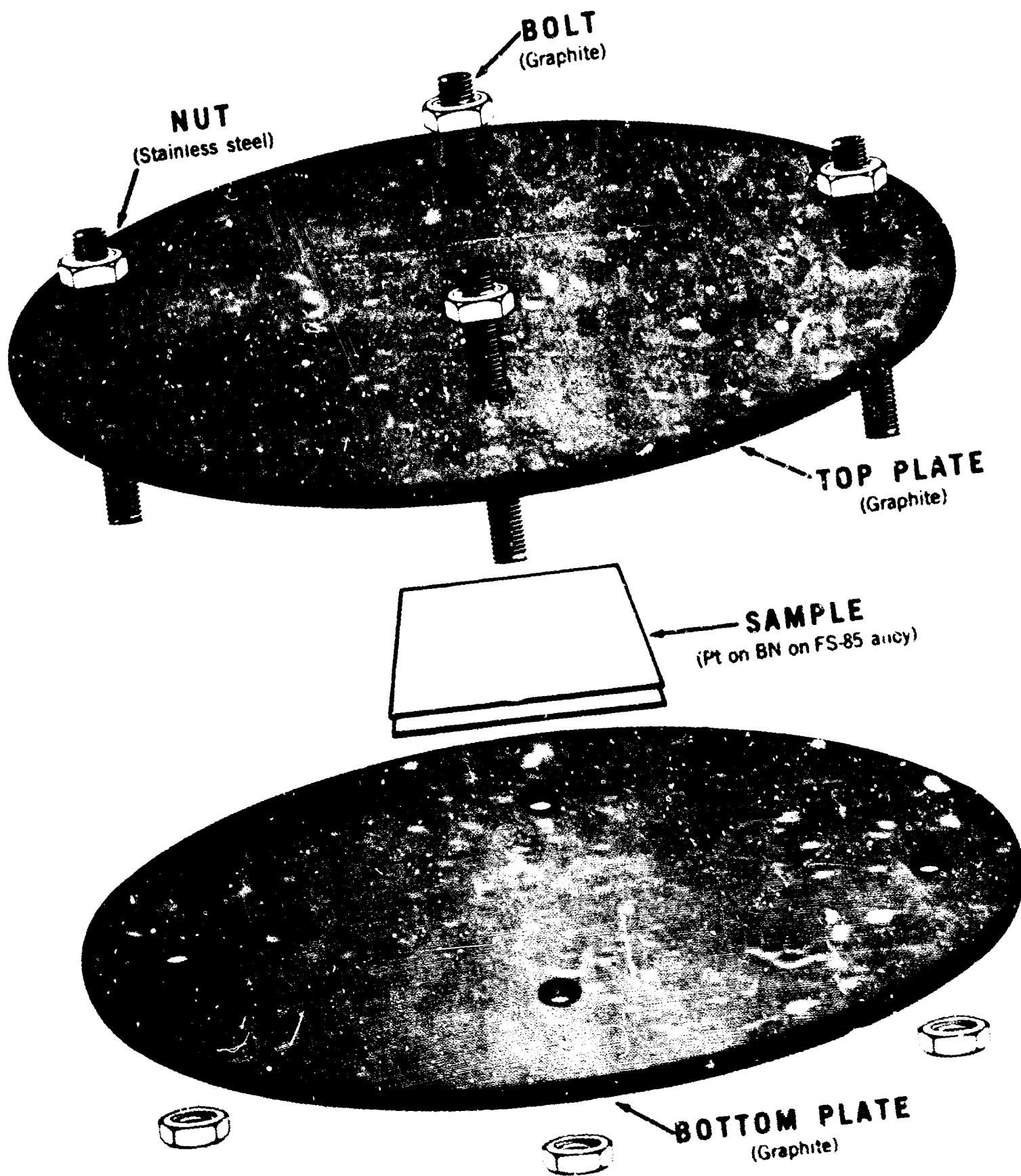


UPPER OUTER FRAME  
 UPPER COVER PLATE  
 PICTURE FRAME  
 FOR SAMPLE  
 LOWER COVER PLATE  
 LOWER OUTER FRAME  
 SAMPLE



**FIGURE 27**  
 Special picture frame setup for testing  
 Boron Nitride coated refractory metal alloys.





**FIGURE 28**  
Setup for hot press bonding platinum coatings onto samples.